

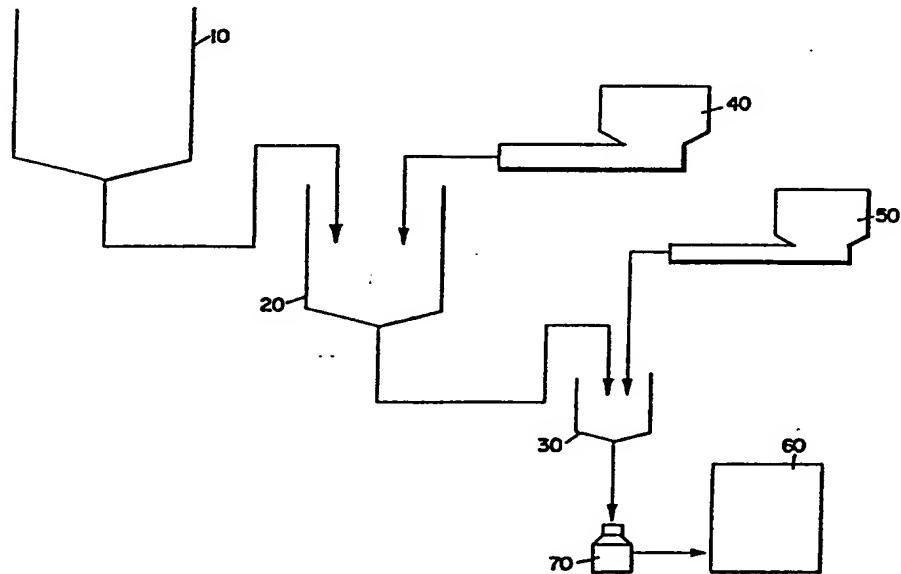


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(71) Applicant:	ECOLAB INC. [US/US]; Ecolab Center, Saint Paul, MN 55102 (US).	Published <i>With international search report.</i>
(72) Inventors:	ROLANDO, John, J. ; 3583 Andrew Drive, Woodbury, MN 55125 (US). MORGANSON, Stephen, A. ; 1564 Ashbury Court, Eagan, MN 55122 (US).	
(74) Agents:	BYRNE, Linda, M. et al.; Merchant, Gould, Smith, Edell, Weiler & Schmidt, 1000 Norwest Center, 55 East Fifth Street, Saint Paul, MN 55101 (US).	

(54) Title: MANUFACTURE OF SOLID, CAST NON-SWELLING DETERGENT COMPOSITIONS



(57) Abstract

A method of manufacturing a solid, cast detergent composition which includes a hydratable chemical capable of combining with water under differing conditions to form at least two distinct hydrated forms which have different densities, such as sodium carbonate, so as to prevent swelling of the cast composition. The process controls the concentration of hydratable chemical, based upon hydratable chemical and water, and the temperature of the mixture throughout the process to minimize formation of the more dense hydrated form of the hydratable chemical.

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**MANUFACTURE OF SOLID, CAST
NON-SWELLING DETERGENT COMPOSITIONS**

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Field of the Invention

The invention relates to the manufacture of solid, cast cleaning compositions which tend to swell after casting. More specifically, the invention relates to the manufacture of solid, cast cleaning compositions which contain a hydratable chemical capable of forming various hydrated forms with significantly different densities so as to avoid transformation of the hydratable chemical between the various hydrate forms after casting.

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Background of the Invention

Solid blocks of alkaline detergent compositions are widely used for household and institutional dishwashing, laundering and general surface cleaning. The manufacturing process employed to produce such solid cast blocks of detergent typically involves heating an aqueous emulsion of the individual components to form a molten melt, blending the molten melt to form a homogeneous mixture, and then casting, cooling and solidifying the homogeneous mixture within a container from which the composition is to be stored, shipped and dispensed.

One component frequently used in the manufacture of solid alkaline detergent compositions is an alkaline builder such as an alkali metal carbonate and/or an alkali metal phosphate. Such alkaline builders are known to be effective for maintaining the detergent solution at an alkaline pH and assisting in the removal of soils from various substrates.

The performance of solid cast detergent compositions is generally enhanced by the inclusion of one or more of the alkaline builders. However, such detergent compositions often exhibit the tendency to swell within the containers after casting. Such swelling frequently results

in deformation of the container to such an extent that the container becomes unsightly and does not conform properly within the dispenser.

Accordingly, a substantial need exists for a procedure which is capable of reducing or eliminating the swelling associated with those solid, cast, detergent compositions containing substantial concentrations of alkaline builders.

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Summary of the Invention

Swelling of a solid, cast, detergent composition which contains a hydratable chemical capable of forming a high density and a low density hydrate may be significantly reduced by cooperatively controlling both the concentration of the dual hydratable chemical, based upon the amount of dual hydratable chemical and water, and the processing temperature so as to minimize formation of the higher density hydrate of the dual hydratable chemical during processing.

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A process for preparing such a non-swelling, solid, cast, detergent composition comprises the step of combining (i) about 10 to about 50 wt% of a dual hydratable alkaline chemical capable of forming a high density and a low density hydrate under different conditions, (ii) about 10 to about 50 wt% water, and (iii) an effective detersive amount of an operative detergent component under conditions which consistently provide a substantially non-swelling cast composition.

Typical dual alkaline hydratable chemicals utilized in detergent compositions include specifically, but not exclusively, sodium carbonate and trisodium phosphate.

When the alkaline hydratable chemical is sodium carbonate the processing temperature of a saturated solution of the sodium carbonate should be maintained below

about 105°F with the concentration of sodium carbonate maintained below 46 wt%. Based upon the processing temperature required to produce a castable composition and the performance benefits obtained by incorporating a major proportion of sodium carbonate, the processing temperature is preferably maintained between about 90° and 105°F with the concentration of the sodium carbonate maintained between about 32 and 46 wt%, based upon the sodium carbonate and water.

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Brief Description of the Drawings

Figure 1 is a schematic diagram depicting a three-stage process useful for practicing the process of the present invention.

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Figure 2 is a phase diagram for the various hydration states of sodium carbonate.

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Detailed Description of the Invention Including a Best Mode

As utilized herein, including the claims, a "dual hydratable alkaline chemical" refers to those hydratable chemicals capable of forming at least one high density hydrate form and at least one low density hydrate form under different conditions wherein the highest density is at least 20% greater than the lowest density.

We have discovered that the post-cast swelling observed in various solid cast detergent compositions is caused by the conversion of certain dual hydratable chemicals present in the composition from a high density hydrated form produced during processing to a low density hydrated form during storage. Based upon this discovery we have developed a process for manufacturing solid, cast detergent compositions which contain a hydratable chemical

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capable of forming a high density and a low density hydrate which minimizes swelling of the cast composition.

As utilized herein, including the claims, the term "wt%" refers to the weight proportion based upon the total weight of the composition unless otherwise specified.

Dual Hydratable Chemicals

Several hydratable chemicals frequently utilized in the formulation of detergent compositions are capable of forming both a high density and a low density hydrate under those conditions commonly encountered in the processing and storage of solid cast detergent compositions. A list of such dual hydratable chemicals includes specifically, but not exclusively, the alkaline detergent builders sodium carbonate and trisodium phosphate. The hydrates and associated densities of these dual hydratable compounds are set forth below.

<u>Compound</u>	<u>Hydrate</u>	<u>Density (g/ml)</u>
Na ₂ CO ₃	anhydrous	2.53
	.H ₂ O	2.25
	.7H ₂ O	1.51
	.10H ₂ O	1.44
Na ₃ PO ₄	anhydrous	2.54
	.H ₂ O	----
	.10H ₂ O	----
	.12H ₂ O	1.62

A Phase diagram depicting the various hydration states of sodium carbonate based upon concentration and temperature is provided as Figure 2. A description of the various regions on the phase diagram is provided below.

<u>Region</u>	<u>Forms</u>
A	Ice
B	Ice + Na ₂ CO ₃ .10H ₂ O

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	C	$\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$
5	D	$\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O} + \text{Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O}$
	E	$\text{Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O}$
	F	$\text{Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O} + \text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$
10	G	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$
	H	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O} + \text{Na}_2\text{CO}_3$
15	I	Na_2CO_3 —
	K	Unsaturated Solutions

Referring to Figure 2, the higher density
 20 anhydrous and/or monohydrate forms of sodium carbonate
 begin to form under saturated conditions, such as
 encountered in the manufacture of solid cast detergent
 compositions, when either (i) the concentration of sodium
 carbonate, based upon sodium carbonate and water, is
 25 greater than about 32 wt% and the temperature of the
 composition exceeds about 105 °F, or (ii) the concentration
 of sodium carbonate, based upon sodium carbonate and water,
 is greater than about 46 wt%, regardless of temperature.
 The presence of the anhydrous and/or monohydrate forms in
 30 the solid cast detergent composition can produce
 perceptible swelling of the composition during storage as
 the higher density anhydrous and monohydrate forms convert
 to the lower density heptahydrate and decahydrate forms
 during storage.

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Process of Manufacture

The process which we have developed to minimize
 the swelling commonly associated with certain solid cast
 detergent compositions includes the step of maintaining the
 40 processing temperature the concentration of the dual

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hydratable chemical within a range which prevents the formation of substantial quantities of the high density form of the dual hydratable chemical. Minimizing formation of the high density form during processing correspondingly 5 reduces the concentration of high density hydratable chemical converting to the low density form during storage which corresponds to a reduction in swelling of the cast composition.

10 Maintaining the concentration of the ~~dual~~ hydratable chemical within the desired range throughout processing is fairly simple once the range is defined. However, maintaining the processing temperature below that temperature at which a significant concentration of the 15 high density hydrate form is developed ($T_{conversion}$) can be complicated by the fact that the minimum temperature necessary for proper processing of typical solid cast detergent compositions containing significant quantities of a dual hydratable chemical ($T_{processing}$) can be only about 10° to about 60°F below $T_{conversion}$ dependent upon the particular dual hydratable chemical and, with respect to sodium carbonate, must be maintained within about a 15°F range (between about 90 and 105°F) to prevent formation of the higher density hydrate forms. In addition, hydration of 25 the dual hydratable chemical is an exothermic reaction which produces substantial quantities of heat which must be controlled in order to prevent the temperature from increasing above $T_{conversion}$. An approximation of the heat of hydration (ΔH) for sodium carbonate, a common dual 30 hydratable chemical found in cast detergent compositions, is set forth below.

<u>Compound</u>	<u>Hydrate</u>	<u>ΔH (kcal/mole)</u>
5 Na_2CO_3	$\cdot\text{H}_2\text{O}$ $\cdot 7\text{H}_2\text{O}$ $\cdot 10\text{H}_2\text{O}$	3.18 16.58 22.13

10 We have discovered that the processing temperature may be closely controlled between $T_{\text{processing}}$ and $T_{\text{conversion}}$ by employing a means for actively cooling the product during processing and providing staged hydration of the dual hydratable chemical. Effective control of the processing 15 temperature requires implementation of both active cooling and staged hydration as each control measure is generally insufficient when use alone to control the temperature within the desired range.

Figure 1 depicts a triple-stage, semi-continuous 20 process for producing a solid cast detergent composition which permits close control over the processing temperature. Broadly, the three-stage process includes the steps of (i) mixing the liquid components of the composition and a first portion of the dual hydratable 25 chemical in a first mix tank 10 to form a base mixture, (ii) mixing a second portion of the dual hydratable chemical and the powdered components of the composition in a first feeder system 40 to form a powder premix, (iii) continuously directing a flow of the base mixture and the 30 powder premix into a second mixing tank 20 equipped with a cooling jacket 25 to produce an intermediate mixture, (iv) continuously directing a flow of the intermediate mixture from the second mixing tank 20 and a final portion of the dual hydratable chemical from a second feeder system 50 35 into a third mixing tank 30 equipped with a cooling jacket 35 to produce the final mixture, (v) casting the final

mixture into molds 70, and (vi) actively cooling the cast mixture in a cooling system 60.

The flow rates into and out from the second tank 20 should be controlled to maintain the intermediate mixture within the second tank 20 for the minimum average time period necessary to result in substantially complete hydration of the dual hydratable chemical within the intermediate mixture before the intermediate mixture is directed into the third tank 30.

In a similar fashion, the flow rates into and out from the third tank 30 should be controlled to maintain the final mixture within the third tank 30 for the minimum average time period necessary to produce a homogeneous final mixture. An extended residence time within the third tank 30 can result in an increase in the viscosity of the final mixture which can interfere with casting of the composition and cause the composition to solidify and adhere to the sidewall of the third tank 30.

Under typical conditions, an average residence time within the second tank 20 of about 20 to about 30 minutes and an average residence time within the third tank 30 of about 2 to about 10 minutes will provide the desired results.

In general, the final mixture should have a viscosity of about 3,000 to 10,000 cps as the mixture is cast into the containers 70. A viscosity of less than about 3,000 cps can permit settling to occur within the composition while a viscosity of greater than about 10,000 cps hampers continued mixing of the final composition within the third tank 30 and complicates casting of the composition into the containers 70. Mixing and casting of the composition can be achieved at viscosities in excess of about 10,000 cps but requires specialized equipment.

During such staged processing, it must be remembered that the concentration of dual hydratable

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chemical must be maintained within that concentration range which prevents formation of the higher density hydrate forms at all times. For example, formation of a sodium carbonate containing detergent composition utilizing the 5 triple-stage process outlined above requires that sufficient sodium carbonate be initially added into the base mixture to produce a base mixture containing at least 32 wt% sodium carbonate, based upon sodium carbonate and water in the composition.

10 The first portion of the dual hydratable chemical is exothermically hydrated by the water in the base mixture within the first mixing tank 10 where the heat generated by the exothermic reaction can be used to bring the temperature of the first mixture up to the processing 15 temperature ($T_{processing}$). Hence, providing other factors do not dictate otherwise, it is preferred to employ sufficient dual hydratable chemical in the base mixture so as to generate sufficient heat of hydration to achieve $T_{processing}$ without exceeding $T_{conversion}$ in the first tank 10. The 20 various factors which affect the amount of dual hydratable chemical which can and should be added into the base mixture include (i) the concentration range of the dual hydratable chemical within which the formation of substantial concentrations of the higher density hydrate 25 form are preventing from developing, (ii) the heat of hydration of the particular hydratable chemical (kcal/mole), (iii) the heat of reaction of any other chemical reaction occurring in the first mixture (kcal/mole), (iv) the moles of first mixture available to 30 absorb the generated heat (moles), (v) the ratio of increase in temperature to increase in heat content for the first mixture ($^{\circ}\text{F}/\text{kcal}$), (vi) the initial temperature of the first mixture ($^{\circ}\text{F}$), (vii) $T_{conversion}$ for the particular dual hydratable chemical ($^{\circ}\text{F}$), and (viii) the capacity of

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the first mixing tank 10 to remove heat from the first mixture.

The second portion of the dual hydratable chemical is exothermically hydrated by the water in the base mixture within the second mixing tank 20. Since the base mixture added to the second mixing tank 20 is only about 10° to about 40°F below $T_{conversion}$, most of the heat generated by addition of the second proportion of the dual hydratable chemical must be promptly removed to prevent the temperature of the intermediate mixture from increasing beyond the 10° to 40°F cushion between the actual temperature (T_{actual}) and $T_{conversion}$. As with addition of the first proportion of the dual hydrated chemical, various factors can affect the amount of dual hydratable chemical which may be added to the intermediate mixture include (i) the heat of hydration of the particular dual hydratable chemical (kcal/mole), (ii) the heat of reaction of any other chemical reaction occurring in the second mixture (kcal/mole), (iii) the moles of intermediate mixture available to absorb the generated heat (moles), (iv) the ratio of increase in temperature to increase in heat content for the intermediate mixture (°F/kcal), (iv) the initial temperature of the base mixture (°F), (v) $T_{conversion}$ (°F), and (vi) the capacity of the second mixing tank 20 to remove heat from the intermediate mixture.

The third portion of the dual hydratable chemical is exothermically hydrated within the third mixing tank 30. As with addition of the second proportion of the dual hydratable chemical, since the intermediate mixture added to the third mixing tank 30 is only about 10° to about 40°F below $T_{conversion}$, most of the heat generated by addition of the third proportion of the dual hydratable chemical must be promptly removed to prevent the temperature of the intermediate mixture from increasing beyond the 10° to 40°F

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cushion between the actual temperature (T_{actual}) and $T_{conversion}$. The same factors considered in determining a suitable quantity of the dual hydratable chemical to be added into the second mixture also apply when determining a suitable 5 quantity of the dual hydratable chemical to be added into the final mixture with the additional factor that all of the dual hydratable chemical must now be incorporated into the final mixture.

The solid cast detergent composition may be cast 10 into a temporary mold from which it is subsequently transferred for packaging in a separate receptacle, or may be cast directly into the receptacle used for shipping and sale. Preferably, the composition is cast directly into the final container in order to eliminate the transfer 15 step.

Several of the factors which influence processing of the composition such as the temperature of the individual components combined to form the base mixture, the temperature of the powdered premix added to the base 20 mixture, the temperature of the third proportion of the dual hydratable chemical added to the intermediate mixture, and the heat removal capacity of the mixing tanks 10,20,30 may be adjusted as necessary. Further, additional stages may be employed in order to reduce the amount of heat 25 generated at any one time.

The process provides excellent control over the swelling associated with various solid cast detergent compositions. The process can typically prevent greater than 3 vol% swelling in at least 95% of the blocks of solid 30 cast detergent composition prepared by the process and is capable of preventing greater than a 2 vol% swelling in at least 98% of the blocks of solid cast detergent composition prepared by the process. Stated in a slightly different manner, the process can typically prevent greater than a 3% 35 decrease in the density of the composition in at least 95%

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of the blocks of cast detergent composition prepared by the process and is capable of preventing greater than a 2% decrease in the density of the composition in at least 98% of the blocks of cast detergent composition prepared by the 5 process. It is noted that a % change in volume does not directly correspond to the same % change in density.

Detergent Components

In addition to the dual hydratable chemical and 10 water, the solid cast detergent composition may include various other detergent components including (i) an alkaline source such as an alkali metal hydroxide, and/or an alkali metal silicate, (ii) a surfactant such as a sulfonate, an ethoxylated alky phenol, and/or a 15 polyoxyalkylene oxide block copolymer, (iii) a chelating agent such as aminopolycarboxylic acid, a condensed phosphate, and/or a polyacrylic acid, (iv) a bleaching agent such as hydrogen peroxide, sodium hypochlorite and/or dichloroisocyanurate, (v) a defoamer such as a defoaming 20 nonionic surfactant, (vi) an enzyme, and (vi) various aesthetically based additives such as dyes and perfumes.

Alkaline Source

An additional detergent component useful in a 25 solid cast detergent composition is an alkaline source. As utilized herein, the term "alkaline source" refers to those alkaline compounds which are known to be useful as builders in detergent compositions for improving soil removal performance. Typical sources of alkalinity include the 30 sodium and potassium hydroxides and the sodium and potassium silicates. Sodium carbonate is also a typical source of alkalinity in detergent compositions but is classified here as a dual hydratable chemical. Such sources of alkalinity are commonly available in either 35 aqueous or powdered form which are both generally useable

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in formulating typical solid cast detergent compositions.

The alkaline source can also assist in solidification of the composition by fixing the free water present in the composition as water of hydration.

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Surfactant

A second detergent component useful in a solid cast detergent composition is a surfactant. Surfactants are those organic compounds which include both a hydrophilic and a hydrophobic moiety on the same molecule and therefore are able to assist in the performance of a detergent composition by enhancing wetting of the substrate, suspending removed contaminants, and various other functions. A wide selection of anionic and nonionic surfactants providing various combinations of characteristics are commercially available from a number of sources. For a detailed discussion of surfactants see Kirk-Othmer, Encyclopdia of Chemical Technology, Second Edition, volume 19, pages 507-592.

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Chelating Agent

A third detergent component useful in a solid cast detergent composition is a chelating/sequestering agent. Generally, chelating agents are those molecules capable of coordinating the metal ions commonly found in natural water and thereby preventing the metal ions from interfering with the functioning of the other deteritive component(s) of the composition. Useful chelating agents include aminocarboxylic acids, condensed phosphates and polyacrylates.

Suitable aminocarboxylic acids include specifically, but not exclusively, N-hydroxyethyliminodiacetic acid, nitrilotriacetic acid (NTA), ethylenediaminetetraacetic acid (EDTA),

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N-hydroxyethyl-ethylenediaminetriacetic acid (HEDTA), and diethylenetriaminepentaacetic acid (DTPA).

Suitable condensed phosphates include specifically, but not exclusively phosphates such as sodium and potassium orthophosphate, sodium and potassium pyrophosphate, sodium tripolyphosphate, and sodium hexametaphosphate. The condensed phosphates can also assist in solidification of the composition by fixing the free water present in the composition as water of hydration.

Suitable polyacrylates include specifically, but not exclusively, polyacrylic acid, polymethacrylic acid, acrylic acid-methacrylic acid copolymers, hydrolyzed polyacrylamide, hydrolyzed polymethacrylamide, hydrolyzed polylamide-methacrylamide copolymers, hydrolyzed polyacrylonitrile, hydrolyzed polymethacrylonitrile, and hydrolyzed acrylonitrile-methacrylonitrile copolymers. Such polyacrylates also function to thicken the detergent composition and thereby assist in solidification. For a detailed discussion of chelating agents/sequestrants, see Kirk-Othmer, Encyclopdia of Chemical Technology, Second Edition, volume 6, pages 1-24.

Bleaches

Many colored materials contain a conjugated chain (a chain of alternating single and double bonds) with a terminal atom or group of atoms which can resonate between two different covalent states. The color of such materials may be destroyed by breaking one of the double bonds in the conjugated chain and/or chemically modifying the terminal atom or atoms so as to prevent resonance. Hence, any chemical compound capable of chemically breaking a conjugated double bond and/or capable of modifying a resonating atom or group of atoms so as to interfere with resonance may be effective as a bleach.

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Broadly, the five categories of bleaching materials include: (i) chlorine, (ii) hypochlorites and chloramines, (iii) hydrogen peroxide and other peroxy compounds, (iv) chlorite and chlorine dioxide, and (v) reducing agents.

Generally, the preferred bleaching agent for use in laundry, warewashing and hard surface cleaners are those bleaches capable of liberating an active halogen species, such as -Cl, -Br, -OCl and -OBr, under those conditions normally encountered in the cleansing processes. Based upon superior bleaching performance and ready availability, the preferred halogen releasing compounds typically include the alkali metal dichloroisocyanurates, chlorinated trisodiumphosphate, the alkali metal hypochlorides, monochloramine and dichloramine.

Bleaching agents are generally deactivated when placed in an alkaline environment such as found within solid cast detergent compositions containing an alkaline source. Accordingly, additional steps may be required to maintain activity of a bleaching agent within a solid cast detergent composition. See, for example, United States Patents Nos. 4,657,784 and 4,681,914.

For a detailed discussion of bleaching agents, see Kirk-Othmer, Encyclopdia of Chemical Technology, Second Edition, volume 3, pages 550-566.

Defoamer

A fifth detergent component useful in a solid cast detergent composition is a defoamant. Typical defoamants are those compounds having a hydrophilic/hydrophobic balance suitable for reducing the stability of foam. The hydrophobicity is typically provided by an oleophilic moiety such as an aromatic alkyl or aralkyl group or an oxypropylene group or chain.

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The hydrophilicity is typically provided by an oxyethylene group or chain or an ester group. The most frequently utilized defoamers include the phosphate esters and the nonionic organic surface-active polymers
5 (surfactants) having both a hydrophobic groups or block and a hydrophilic ester-groups or block such as a nonylphenol ethoxylate. A discussion of defoaming nonionic surfactants may be found in United States Patents Nos. 3,048,548 (Martin et al), 3,334,147 (Brunelle et al), and
10 3,442,242 (Rue et al).

Enzyme

A sixth detergent component useful in a solid cast detergent composition is an enzyme effective for catalyzing the break down of various contaminating compounds commonly found on the substrate(s) to be cleaned. Commonly employed enzymes include the proteinases which are capable of catalyzing the hydrolysis of proteinaceous materials, amylases which are capable of catalyzing the hydrolysis of polysaccharides, and lipases which are capable of catalyzing the hydrolysis of fats.

In addition to those components previously described, other conventional detergent components may be incorporated into the detergent composition so long as they do not meaningfully interfere with solidification of the composition nor with maintenance of the processing parameters within those established for preventing the formation of the more dense hydrate form of dual hydratable chemical. Such components may include such compounds as bactericides, brightening agents, antiredeposition agents, inorganic salts, dyes and fragrances.

Broadly, a solid cast detergent composition based upon sodium carbonate may be prepared in accordance with the three-stage process described above by (-) including in
35 the base mixture, with preferred ranges provided in

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brackets, (a) about 10 to 20 wt% [12 to 16 wt%], based upon the final mixture, sodium carbonate, (b) about 20 to 40 wt% [25 to 35 wt%], based upon the final mixture, water, (c) about 0 to 5 wt% [2 to 5 wt%], based upon the final
5 mixture, polycarboxylic acid, (d) about 0 to 5 wt% [1 to 3 wt%], based upon the final mixture, sodium hydroxide, and (e) about 0 to 5 wt% [2 to 5 wt%], based upon the final mixture, surfactant; (-) including in the powder premix, with preferred ranges provided between brackets, (aa) about
10 4 to 10 wt% [5 to 6 wt%], based upon the final mixture, of a second portion of the sodium carbonate, (bb) about 0 to 50 wt% [30 to 40 wt%], based upon the final mixture, sodium tripolyphosphate, and (cc) about 0 to 5 wt% [1 to 3 wt%], based upon the final mixture, enzyme; and (-) adding about
15 1 to 5 wt% [1 to 3 wt%] of a third portion of the sodium carbonate in the third stage.

The sodium tripolyphosphate is preferably added in hydrated form to prevent the sodium tripolyphosphate from competing with the sodium carbonate for water of
20 hydration within the composition.

Dispensing

The solid cast detergent composition may be conveniently dispensed from a spray-type dispenser such as
25 those disclosed in United States Patents Nos. 4,426,326, 4,569,780, 4,569,781 and 4,687,121. Briefly, spray-type dispensers function by supporting a solid block of material upon a screen above a spray nozzle and directing a water spray from the spray nozzle through the screen and into
30 contact with an exposed surface of the solid block so as to dissolve a portion of the solid block of material and form a concentrated solution which is immediately directed to the point of use.

With respect to a solid cast detergent composition
35 based upon sodium carbonate, the concentration of sodium

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carbonate in the product between the permissible range of 32 and 46 wt% can have a substantial effect upon the dispensing rate of the product in a spray-type dispenser. Product having a higher concentration of sodium carbonate 5 is less water soluble and hence dispenses slower while product having a lower concentration of sodium carbonate is more water soluble and hence dispenses faster.

The present invention will be further understood by reference to the following specific Examples which are 10 illustrative of the method of producing a solid, cast detergent composition of this invention. Since many variations of the invention may be made without departing from the spirit and scope of the invention, the breadth of the invention resides in the claims hereinafter appended.

¹⁹
Procedure
Runs # 1-4

A base solution containing the components set forth in Table 1 was prepared in a 1000 gallon mixing tank equipped with a variable speed, pitched blade turbine agitator and a water cooling jacket. The temperature of the base solution within the 1000 gallon tank was maintained within the temperature range set forth in Table 2.

A blend of the powdered components set forth in Table 1 was prepared in a Ribbin Blender. The powdered blend was generally maintained at about room temperature which is set forth in Table 2.

The base solution and the powdered premix were then fed continuously into a 150 gallon tank equipped with a variable speed, pitched blade turbine agitator and a water cooling jacket at the flow rates set forth in Table 2 to produce an intermediate mixture. The flow rate of the base mixture was calculated by measuring the weight loss from the 1000 gallon tank over time. The flow rate of the powdered premix was established by feeding the powdered premix through an Acrison™ powder feeder. The two streams reacted exothermically as the dual hydratable chemical in the powdered premix was hydrated by the water in the base mixture. The temperature of the intermediate mixture within the 150 gallon tank was maintained within the temperature range set forth in Table 2 by actively cooling the intermediate mixture with the cooling jacket. The volume of intermediate mixture within the 150 gallon tank was maintained within the range set forth in Table 2 throughout the process until the supply of base solution and/or powdered premix was depleted.

The intermediate solution and a third stage addition of the dual hydratable chemical, in an amount set

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forth in Table 1, were then feed continuously into a 40 gallon tank equipped with a variable speed, pitched blade turbine agitator and a water cooling jacket at the flow rates set forth in Table 2 to produce a final mixture. The 5 flow rate of the intermediate mixture was estimated by maintaining a constant volume of final mixture in the 40 gallon tank and measuring the flow rate of the final mixture out of the 40 gallon tank. The flow rate of the dual hydratable chemical fed in the third stage addition 10 was established by feeding the dual hydratable chemical through an Acrison™ powder feeder. The two streams reacted exothermically as the dual hydratable chemical was hydrated by the water in the base mixture. The temperature of the final mixture within the 40 gallon tank was maintained 15 within the temperature range set forth in Table 2 by actively cooling the final mixture with the cooling jacket. The volume of final mixture maintained within the 40 gallon tank was within the range set forth in Table 2 throughout the process until the supply of intermediate solution was 20 depleted.

The final mixture in the 40 gallon tank was cast into approximately 8 pound blocks within a polypropylene bottle utilizing a Simplex™ piston filler. The cast product was then cooled within a cooling water spray 25 tunnel. The temperature of the cooling water and the duration of cooling within the tunnel are set forth in Table 3.

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Nomenclature

	NA ₂ CO ₃	Sodium Carbonate or Dense Ash.
5	NaOH-bead	Solid sodium hydroxide in bead form.
	PAA	A polyacrylate having an average molecular weight of about 4,500 manufactured by Ecolab, Inc. of St. Paul, Minnesota.
10	NPE 9.5	Nonylphenol ethoxylate having an average of 9.5 ethylene units per nonylphenol.
15	LAS 90F [®]	A 90% active flaked linear alkyl benzene sulfonate sodium salt available from Witco Chemical, Stepen Chemical, Pilot Chemical and Vista Chemical.
20	STPP	Sodium tripolyphosphate.
	Esp 6.0T [®]	A protease enzyme available from Novo.

I claim:

1. A process comprising the steps of (-) preparing a solid cast detergent composition which is prone to swelling after casting and includes a dual hydratable chemical capable of forming a high density hydrate form and a low density hydrate form under different conditions, and (-) maintaining the processing conditions employed in preparing the solid cast detergent composition so as to minimize the concentration of the high density form of the dual hydratable chemical within the composition at the time of casting and thereby reduce swelling of the solid cast detergent compositions after casting.
2. The process of claim 1 wherein the dual hydratable chemical is selected from the group consisting of sodium carbonate and sodium phosphate.
3. The process of claim 1 wherein the dual hydratable chemical is sodium carbonate and the step of maintaining the processing conditions employed in preparing the solid cast detergent composition so as to minimize the concentration of the high density form of the dual hydratable chemical within the composition at the time of casting includes (-) maintaining the concentration of the sodium carbonate between that necessary to produce a saturated solution and 46 wt%, based upon the amount of sodium carbonate and water, and (-) maintaining the processing temperatures after incorporation of the dual hydratable chemical between that necessary to achieve a castable composition and about 105°F.

4. The process of claim 1 wherein the dual hydratable chemical is sodium carbonate and the step of maintaining

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the processing conditions employed in preparing the solid cast detergent composition so as to minimize the concentration of the high density form of the dual hydratable chemical within the composition at the time of casting includes (-) maintaining the concentration of the sodium carbonate between about 32 and 46 wt%, based upon the amount of sodium carbonate and water, and (-) maintaining the processing temperatures after incorporation of the dual hydratable chemical between about 90°F and about 105°F.

5. The process of claim 1 wherein the step of preparing a solid cast detergent composition under conditions which minimize swelling of the composition after casting comprises the step of preparing the solid cast detergent composition under conditions which result in a swelling of less than 3 vol% in at least 95% of the blocks of cast detergent composition.

6. The process of claim 1 wherein the step of preparing a solid cast detergent composition under conditions which minimize swelling of the composition after casting comprises the step of preparing the solid cast detergent composition under conditions which result in a swelling of less than 2 vol% in at least 98% of the blocks of cast detergent composition.

7. The process of claim 1 wherein the step of preparing a solid cast detergent composition under conditions which minimize swelling of the composition after casting comprises the step of preparing the solid cast detergent composition under conditions which result in at least 95% of the blocks of cast detergent composition maintaining a substantially constant density after casting.

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8. The process of claim 1 wherein the step of preparing a solid cast detergent composition under conditions which minimize swelling of the composition after casting comprises the step of preparing the solid cast detergent composition under conditions which result in at least 98% of the blocks of cast detergent composition having less than a 2% decrease in the density of the composition after casting.

9. The process of claim 1 wherein the dual hydratable chemical is sodium carbonate and the step of maintaining the processing conditions employed in preparing the solid cast detergent composition so as to minimize the concentration of the high density form of the dual hydratable chemical comprises the step of maintaining the processing conditions employed in preparing the solid cast detergent composition so as to minimize the concentration of anhydrous sodium carbonate and sodium carbonate monohydrate.

10. A process for preparing a solid, cast, non-swelling, detergent composition comprising the steps of:

- (a) forming a base mixture which includes (i) a first portion of a hydratable chemical capable of combining with water under differing conditions to form at least two distinct hydrated forms which have different densities, and (ii) water;
- (b) adding to the base mixture a powder premix which includes at least (i) a second portion of the dual hydratable chemical, and (ii) an operative detergent component to form an intermediate mixture;

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- (c) adding a third portion of the dual hydratable chemical to the intermediate mixture to form a final mixture; and
- (d) casting the final mixture to form the solid, cast detergent composition;

wherein the processing conditions are maintained throughout the process so as to produce a final mixture which is substantially free of the high density hydrated form of the dual hydratable chemical.

11. The process of claim 10 wherein the step of casting the final mixture comprises the step of casting the final mixture when the final mixture has a viscosity of about 3,000 to about 10,000 cps.

12. The process of claim 10 wherein the step of adding a powder premix to the base mixture comprises the step of adding a mixture of a second portion of the dual hydratable chemical and sodium tripolyphosphate hexahydrate to the base mixture.

13. The process of claim 10 wherein (i) the base mixture and the powder premix have a mixing residence time of about 20 to about 30 minutes before addition of the third portion of the dual hydratable chemical, and (ii) the intermediate mixture and third portion of the dual hydratable chemical have a mixing residence time of about 2 to about 10 minutes before casting of the final mixture.

14. The process of claim 10 wherein the step of forming a base mixture which includes a dual hydratable

chemical and water comprises the step of forming a base mixture which includes sodium carbonate and water.

15. The process of claim 10 wherein the step of forming a base mixture which includes at least a dual hydratable chemical and water comprises the step of forming a base mixture which includes at least:

- (a) about 10 to about 20 wt%, based upon the final mixture, sodium carbonate which is capable of forming at least one high density hydrate form and at least one low density hydrate form under different conditions;
- (b) about 20 to about 40 wt%, based upon the final mixture, water;
- (c) about 2 to about 5 wt%, based upon the final mixture, polycarboxylic acid;
- (d) about 1 to about 3 wt%, based upon the final mixture, sodium hydroxide; and
- (e) about 2 to about 5 wt%, based upon the final mixture, surfactant.

16. The process of claim 10 wherein the step of adding a powder premix to the base mixture comprises the step of adding to the base mixture a mixture which includes at least (i) about 4 to 10 wt%, based upon the final mixture, of a second portion of the dual hydratable chemical, (ii) about 20 to 40 wt%, based upon the final mixture, hydrated sodium tripolyphosphate, and (iii) about 1 to 3 wt%, based upon the final mixture, enzyme.

17. A process for preparing a solid, cast, non-swelling, detergent composition comprising the steps of:

- (a) forming a base mixture which includes at least a first portion of sodium carbonate

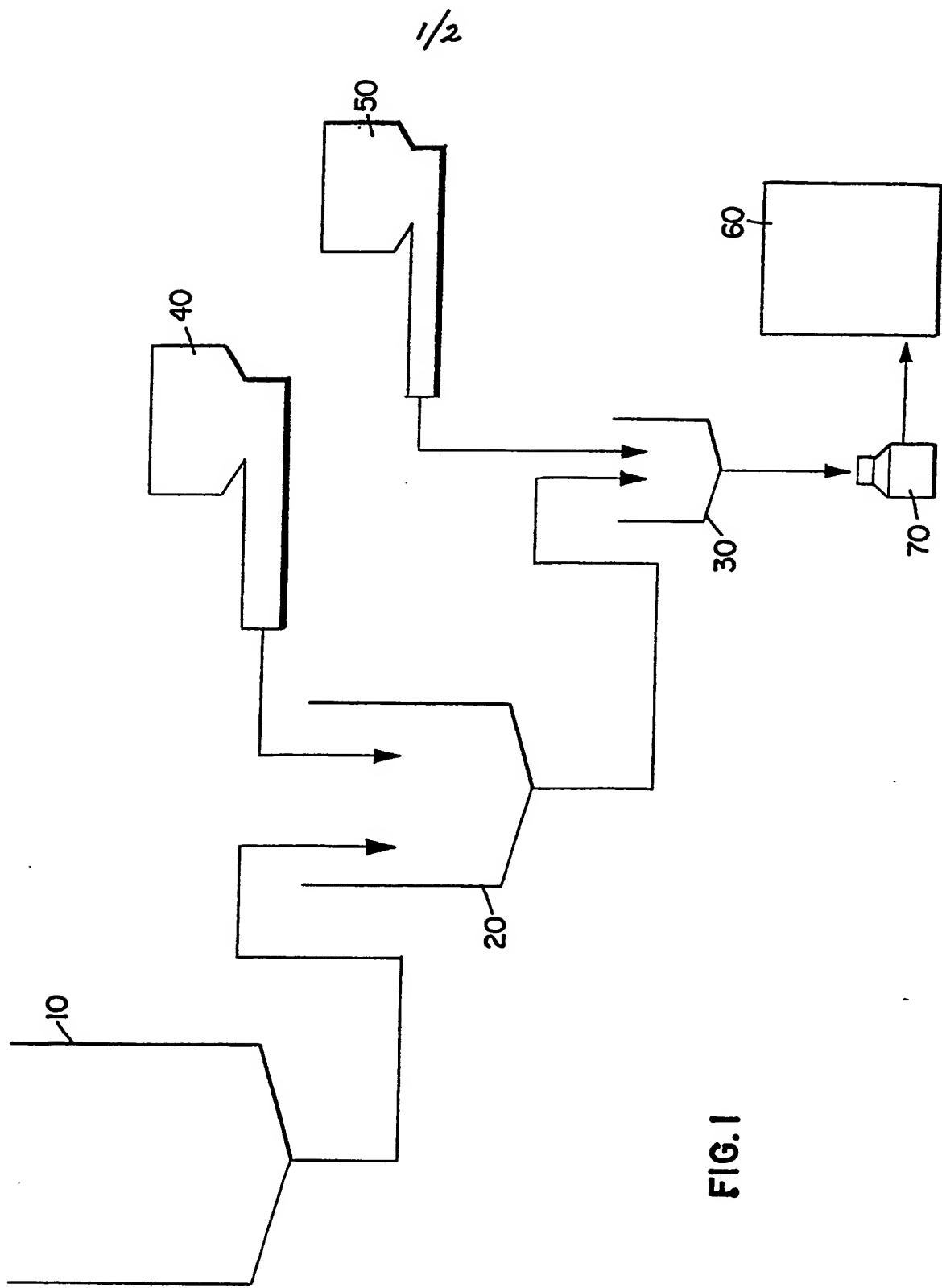
and water wherein the concentration of sodium carbonate in the base mixture is maintained between about 32 and 46 wt%, based upon the amount of sodium carbonate and water, and the temperature of the base mixture is maintained between about 90° and 105°F;

- (b) adding to the base mixture a powder premix which includes at least a second portion of the dual hydratable chemical and an operative detergent component to form an intermediate mixture wherein the concentration of sodium carbonate in the intermediate mixture is maintained between about 32 and 46 wt%, based upon the amount of sodium carbonate and water, and the temperature of the intermediate mixture is maintained between about 90° and 105°F;
- (c) adding to the intermediate mixture a third portion of the sodium carbonate to form a final mixture wherein the concentration of sodium carbonate in the final mixture is maintained between about 32 and 46 wt%, based upon the amount of sodium carbonate and water, and the temperature of the final mixture is maintained between about 90° and 105°F; and
- (d) casting the final mixture to form the solid, cast detergent composition;

whereby the final mixture is substantially free of the anhydrous and monohydrate forms of sodium carbonate.

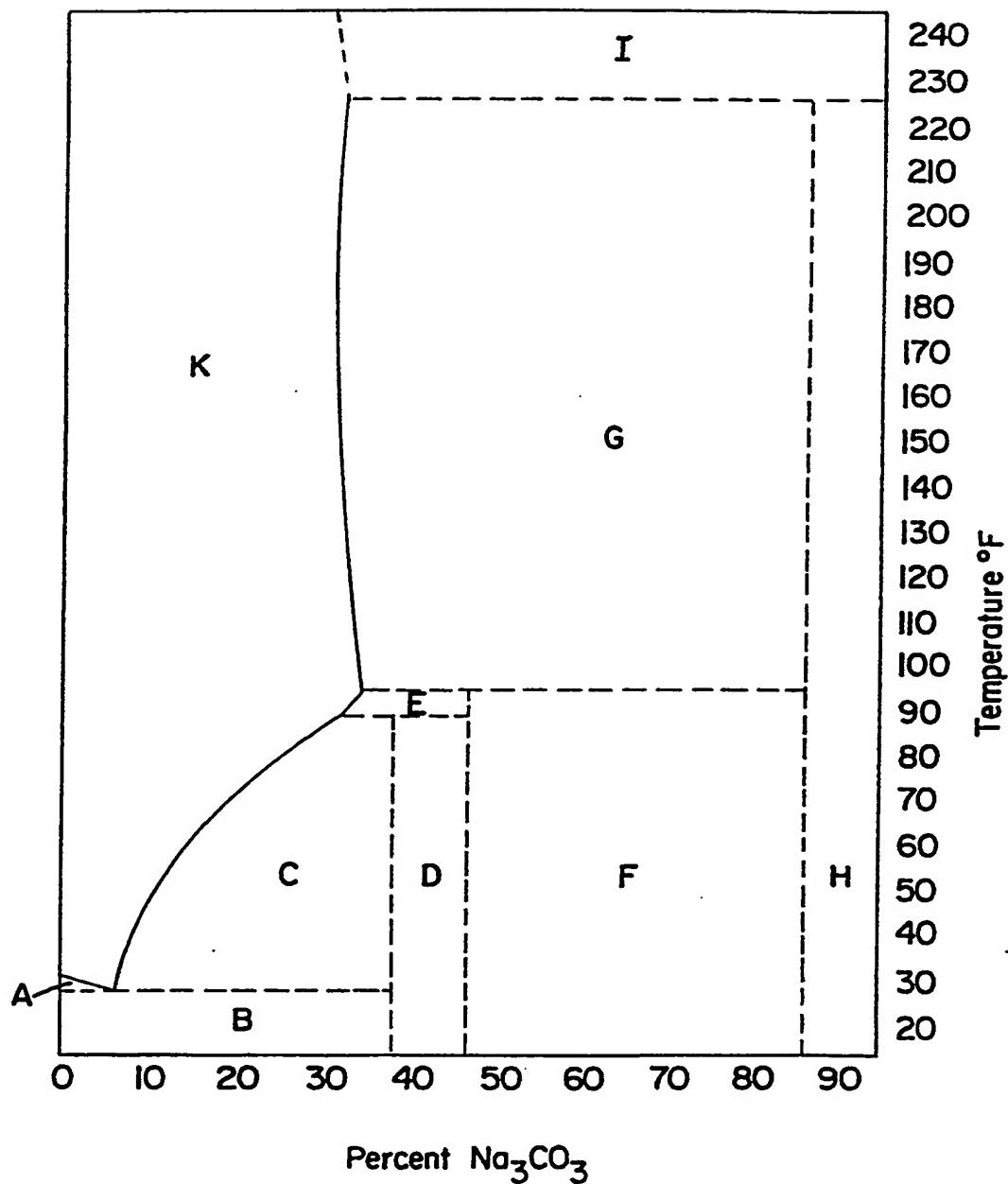
18. The product obtained by the process of claim 1 wherein the cast composition is consistently non-swelling.

19. The product obtained by the process of claim 2 wherein the cast composition is consistently non-swelling.
20. The product obtained by the process of claim 3 wherein the cast composition is consistently non-swelling.
21. The product obtained by the process of claim 4 wherein the cast composition is consistently non-swelling.
22. The product obtained by the process of claim 5 wherein the cast composition is consistently non-swelling.
23. The product obtained by the process of claim 7 wherein the cast composition is consistently non-swelling.
24. The product obtained by the process of claim 9 wherein the cast composition is consistently non-swelling.
25. The product obtained by the process of claim 10 wherein the cast composition is consistently non-swelling.
26. The product obtained by the process of claim 13 wherein the cast composition is consistently non-swelling.
27. The product obtained by the process of claim 14 wherein the cast composition is consistently non-swelling.
28. The product obtained by the process of claim 15 wherein the cast composition is consistently non-swelling.
29. The product obtained by the process of claim 16 wherein the cast composition is consistently non-swelling.
30. The product obtained by the process of claim 17 wherein the cast composition is consistently non-swelling.



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FIG. 2



ANHANG

zum internationalen Recherchenbericht über die internationale Patentanmeldung Nr.

ANNEX

to the International Search Report to the International Patent Application No.

ANNEXE

au rapport de recherche international relatif à la demande de brevet international n°

PCT/US91/05162 SAE50624

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